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The Flow Method of Ultramicroscope Measurement of the Particle Concentration of Aerosols and Other Dispersion Systems

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The existing ultramicroscopic methods of making particle counts of aerosols and other dispersion systems in general have real shortcomings, which give rise to errors in our determinations, including some of a systematic nature, and cause much lost time in making observations, particularly when we are dealing with small particle concentrations.

The idea of the method of FLOW ULTRAMICROSCOPY*, as worked out by us, is the making of a particle count in a continuous stream of aerosol flowing in a direction parallel to the line of sight,** with the particles traversing a zone of illumination in a set time. If the total observed number of the little flashes in the field of view made by particles crossing the zone of illumination is divided by the volume of the aerosol flowing over the field, we obtain the particle concentration. Figure 1 is a diagram of the apparatus constructed by ourselves for this purpose.

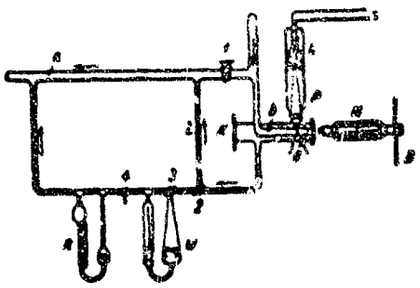

Fig. 1

The main aerosol stream goes through the large pipe a, by-passing the gas cell K. To give the gas cell a preliminary "washing through" with the test-aerosol, the cock 1 is closed and the aerosol stream is channeled by means of the three-way cock 2 through the gas cell K and pipe L. Before making the count, cock 2 is turned so that the aerosol, after flowing through the gas cell, passes through micro-valve 4 and the capillary tube of flow-meter R. The micro-valve and flow-meter permit one to regulate and measure the volumetric velocity of flow of the aerosol through the gas cell. In the gas cell the aerosol first flows through the inner tube b and then through the space c between the inner tube and the outer cylindrical wall of the cell. The cell is made of glass, with the inner tube blackened except for an illumination window. The dark-field illumination is provided by an automobile lamp, an accumulator-fed incandescent bulb with the filament heated somewhat above normal. The light from this bulb passes through lens P and is focused on the central part of the cell. The aerosol particles are observed as they pass through the zone of illumination by means of microscope M, the eyepiece of which is fitted with a revolving diaphragm D having a series of openings ranging in diameter from 0.1 to 10 mm. After the gas cell has been "washed through", the three-way cock 3 is turned so as to pass the aerosol stream into volume gauge /W/ and simultaneously one starts to count the flashes observable in the field of the microscope.

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* This method forms the content of a Certificate of Authorship No. 325565 issued to us on the basis of our Declaration made on the 19th of June, 1944. [This refers to a patent or copyright registration. Tr.]

** Particle-counting in a transverse stream is considerably less convenient.

The count is stopped either when a certain number of flashes has been recorded (for instance, one hundred) or upon a signal indicating that a certain volume has passed. At the same time as the count is stopped the volume gauge is shut off by turning cock 3. The volume which has passed is then calculated from the rise in the level of the liquid in the straight leg of volume gauge M.

The particle concentration may be calculated by the formula

$$n = \frac{N}{V} = a\frac{N}{W} \tag{1}$$

where N is the total number of flashes observed, V is the volume which has passed through the count area, i.e. the area outlined in the visual field of the microscope, W is the volume which has passed through the gas cell passage b, W being related to V by the ratio coefficient a. For linear flow of the aerosol through passage b of the gas cell (which was always the case in our tests), the parabolic diametral distribution of velocities over the cross section of the passage works out to the formula

$$a = \frac{D^2}{d^2\left[2-(d^2/D^2)\right]} \tag{2}$$

where D is the diameter of the inner passage of the gas cell and d is the diameter of the count area.

Let us now consider the precision, reproducibility, sensitivity and performance of the apparatus. A fundamental source of error in this method is variations in the number of particles per given volume. This is a difficulty which is inherent and inevitable in all ultramicroscopic methods. The arithmetical mean error thus introduced in measurements of the concentration is equal to

$$\Delta n/n \cong 0.8/\sqrt{N} \tag{3}$$

where N is the number of flashes counted.

To reduce the error $\Delta n/n$ to 5% it is sufficient to take N = 400, which would require a four-minute count at a counting speed of about 100 per minute. The counting speed may be adjusted to any value we choose; we may change the velocity of aerosol flow through micro-valve N and we may vary the count area (either by means of the eye-piece diaphragm or by changing the objective). In making counts of particle concentrations of the order of 10^7 cm^{-3} it is necessary to use an eye-piece diaphragm opening of about 0.1 mm diameter in conjunction with a 10x or 20x objective and to have a velocity of the aerosol-flow along the axis of the gas cell not greater than 1 mm/sec. At the same time it is necessary to cut down on the zone of illumination by means of an interposed diaphragm, until the thickness of the said zone is less than the focal depth of the microscope, which amounts to a few hundredths of a millimeter. Under these conditions we almost eliminate the chance of there being more than one particle in the count area at any one time. The flashes will follow each other separately and will be seen against a dark background. Since it is easy to eliminate error in the flash-count N, and since we have discarded the idea of measuring the count-volume, the inexact technique of the older ultramicroscopic methods, and instead are measuring the volume flowing through the apparatus, which can be easily carried out with great precision, all sources of error other than the variation in particle-distribution are of no practical importance in comparison. This has been confirmed in numerous actual counts.

Thus for instance we determined the value of n for oil fog ten times in succession (with N = 50 for each count) and obtained the following figures (Table I):—

TABLE I

PARTICLE CONCENTRATION IN OIL FOG

Counts					
N	W, cm^3	$\alpha \times 10^{-5}$	$n\ cm^{-3} \times 10^{-7}$	Deviation from mean value Δn	% Deviation from mean, $\Delta n/n_{mean}$
50	0.50	2.67	2.67	+0.26	10.8
50	0.50	2.67	2.67	+0.26	10.8
50	0.70	2.67	1.91	−0.50	20.7
50	0.60	2.67	2.22	−0.19	7.9
47	0.50	2.67	2.67	+0.10	4.1
50	0.50	2.67	2.67	+0.26	10.8
50	0.50	2.67	2.67	+0.26	10.8
50	0.50	2.67	2.67	+0.26	10.8
50	0.60	2.67	2.23	−0.18	7.5
50	0.70	2.67	1.91	−0.50	20.7
			2.41		11.5

Here we obtain $\Delta n/n_{mean} = 11.5$ as the experimental value of the error, which is quite near to the theoretical value $0.8\sqrt{17/N} \cdot 100 = 11.3\%$.

In most ultramicroscopic techniques, the most important advantage of the flow method is the great saving in time in the measurement of small particle concentrations, a fact which will be evident from the following calculation.

Let the count volume (count area S multiplied by the thickness h of the zone of illumination) be identical in both the systems we are comparing. With the old methods, the contents of the count volume are changed approximately once a second, while with the flow method this change takes place in a time t, namely the time it takes each aerosol particle to flit across the zone of illumination, a time equal to the duration of the flashes. Experiments show that we can take t = 0.01 second without interfering with the visibility of the flashes, a fact which is in agreement with the findings of visual physiology in the matter of the sensitivity of the eye to momentary illumination. Under these circumstances the flow method speeds up the particle count at least a hundred times or in other words permits us, in the same space of time and for the same variational error, to count a particle concentration at least a hundred times smaller. Thus a concentration of the order of 1 cm^{-3} may be easily measured in a time of the order of one minute, with a mean error of the order of 20%. Let us briefly enumerate the fundamental advantages of the flow method in the determination of particle concentrations, as follows:—

(1) A reduction (by 100 times or more) of the time expended in measuring small particle concentrations; that is, we are enabled to measure, with the same expenditure of time, particle concentrations of one hundredth the density or less.

(2) The technique of measuring the volume of flow of the aerosol with a separate volume gauge enables us to avoid the difficulties and errors which arise if we are under the necessity of exactly delimiting and precisely determining the count-volume; in particular this technique obviates the indeterminateness of the count-volume caused by secondary diffusion.

(3) It is not necessary to list and total up a series of readings.

(4) As compared with the method of counting particles in a physically enclosed volume, the flow method has these advantages: use of a simpler gas cell, absence of dispersed light from the surfaces enclosing the count-volume, and absence of error due to the settling of aerosol particles on these surfaces.

(5) The small volume of the cell and the avoidance of periodical interruptions of the aerosol flow (inevitable with the old method) preclude errors due to the settling, coagulation, condensation, and evaporation of aerosol particles.

(6) The flow method gets rid of all errors connected with sedimentation and with the Brownian movement, factors which with the usual methods and when counts are being made of high initial concentrations of highly dispersed systems are capable of introducing quite serious errors.

(7) There is no need of high magnification even in the measurement of high concentrations; thus we do not need such exact definition and consequently can be less particular about the optical system of the microscope.

(8) The flow method reveals a new possibility of fundamental nature: it may lead to an automatic method for aerosol particle counts.

(9) The flexibility of the flow method makes it easy to range over a wide scale of concentrations, from 3.10^7 or 4.10^7 down to 1 or 2 per cm^3.

(10) The apparatus as constructed by us is portable and hence may be used even under non-laboratory conditions.

The flow method will inevitably find very diversified applications in connection with a number of scientific problems, especially in the study of rapidly occurring processes in aerosols, since it is possible to measure particle concentrations in a swiftly flowing stream of the aerosol immediately after the formation of the latter, or immediately after it has been acted upon in some way (for instance by an electrostatic charge). In our laboratory in particular the flow method has been used by I.S. ARTEMOV* for studying the effect of foreign vapors on aerosol coagulation. In Figure 2 we give a sample coagulation graph (particle volume as a function of time) showing that the coagulation speed of oil fog is independent of the presence of oleic acid vapor (compare black circlets with open circlets)

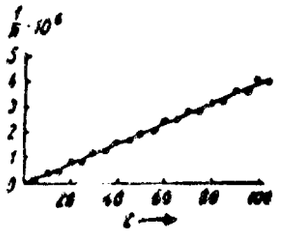

Fig. 2

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* I.S. ARTEMOV, Journal of Physical Chemistry [USSR], 20, 559, (1946).